DETERMINATION OF THE NFPA REACTIVITY RATING

OF AUTOMATE YELLOW 96

BY DIFFERENTIAL SCANNING CALORIMETRY

TO : U.S. Chemical Safety & Hazard Investigation Board
2175 K Street N.W.
4th Floor
Washington, DC 20037

F.A.O.: Mr. David Heller

Prepared By .... **Reviewed By ....**

Scott M. Ferguson
Laboratory Technician

Charles Gilbert
Sr. Chemist

Approved By ....

Richard C. Wedlich
Sr. Process Safety Specialist

FOR AND ON BEHALF OF CHILWORTH TECHNOLOGY, INC.

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1. INTRODUCTION

The U.S. Chemical Safety and Hazard Investigation Board is concerned with the hazards associated with the Automate Yellow 96. Specifically, the board requires the accurate determination of the material's reactivity rating.

The present study was undertaken to determine the appropriate NFPA reactivity rating via Differential Scanning Calorimetry and UN/DOT 4.3 'Dangerous When Wet' testing. This report addresses Differential Scanning Calorimetry performed on the sample.

Mr. David Heller at the U.S. Chemical Safety and Hazard Investigation Board is the U.S. Chemical Safety and Hazard Investigation Board's representative for this project.
Mr. Richard Wedlich at Chilworth Technology, Inc. supervised the analytical work performed by the Chemical Process Evaluation group at Chilworth, including Mr. Scott M. Ferguson.

2. SCOPE OF THE ASSESSMENT

This assessment follows discussions between Mr. David Heller (U.S. Chemical Safety & Hazard Investigation Board) and Mr. Richard Wedlich (Chilworth Technology, Inc.).

A proposed scope of work included DSC Analysis, Dangerous When Wet testing (per UN/DOT Division 4.3), BAM Fallhammer (impact sensitivity testing), Koenen tube (confinement testing) and BAM Friction (friction sensitivity testing) on the material provided by Mr. Heller. The aim of the assessment is to determine the appropriate NFPA reactivity rating for the sample.

Safety in chemical manufacturing requires that all possible operational hazards, i.e. the presence and possible ignition of flammable atmospheres, and chemical reaction hazards are evaluated and that a suitable basis for safe operation is determined and implemented.

Should the reaction conditions or plant details be changed (e.g. temperature, times, reaction concentrations, scale, materials of construction or maloperations not covered by this assessment) then consideration should be given to re-assessment of the process.

3. DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Test Objective

To quantify the onset temperature and magnitude of any thermal activity in a sample while heating it over a given temperature range.

Test Principle

Differential Scanning Calorimetry is a thermal technique in which the temperature of a sample, compared with the temperature of a thermally inert material, is recorded as a function of the furnace temperature as the sample is heated or cooled at a uniform rate. Temperature changes in the sample are due to endothermic or exothermic enthalpic transitions such as that caused by chemical reactions or phase changes.

Test Apparatus

The test was performed in a Mettler® DSC, model 821e, Thermal Analysis System. This system is computer controlled and allows for immediate online analysis of the data upon completion of the test.

Schematic diagrams of the calorimeter and typical experimental trace are shown in **Figure 1**. The left hand side of Figure 1 shows the cross section of the furnace used to heat sample (located in the left cup) and reference (located in the right cup) through the desired experimental temperature range. In the present case, an empty cup was used as a reference. A differential thermocouple is used to measure the temperature difference between the sample and reference as a function of time and temperature. When the sample undergoes a thermal transition a non-zero differential is created (?T) between the sample and reference temperatures. This differential over time is converted into a power. A plot of power versus temperature is shown on the right hand side of Figure 1 for an exothermic transition.

Sensitivity

The DSC can accurately measure power changes of 0.07 microwatts.

Test Method

The computer and DSC were turned on, user logged in and DSC program started. The sample was weighed to the tenth milligram into an aluminum DSC pan, top placed on the pan and the pan sealed shut. A final weight on the sealed pan was taken and the pan was placed into the automatic sample loader. The run parameters were programmed into the DSC (i.e. temperature range and rate of heating) and the run started. Data was collected throughout the run and plotted in real time on the computer.

Data Interpretation

The temperature differential between the sample and the reference is converted to heat units using a standard calibration curve that is stored in the microprocessor unit. This heat value is next converted to a power by dividing by the run time. It is the power vs. temperature (or time) plot that constitutes a thermogram. The interpretation of the thermogram is generally straightforward; an exotherm is shown as an upward deviation in the baseline while an endotherm is shown as a downward deviation in the baseline. The deviation from the baseline determines the onset temperature for the transition while the peak area determines the enthaply change.

4. RESULTS

Testing Objective

To accurately determine the onset temperature for exothermic activity.

Sample Information

Company name	:	U.S. Chemical Safety and Hazard Investigation Board
Source of Samples	:	Provided by the client
Test Date	:	1/28/00
Operator	:	S. Ferguson
Sample Conditions	:	Sample was run in the 'as-received' condition

Results

Differential Scanning Calorimetry	**Run 01**	**Run 02**
Sample Wt.	23.3100 mg	5.8400 mg
Scan Rate	20° C/min	Variable - 5° C/min @ onset
T_{START}	25° C	25° C
T_{END}	330° C	300° C
T_0 – exotherm*	240° C	239° C
T_0 – endotherm	N/A	N/A

*Taking the onset temperature as the temperature at which the leading edge of the exotherm peak first deviates upward from the baseline.

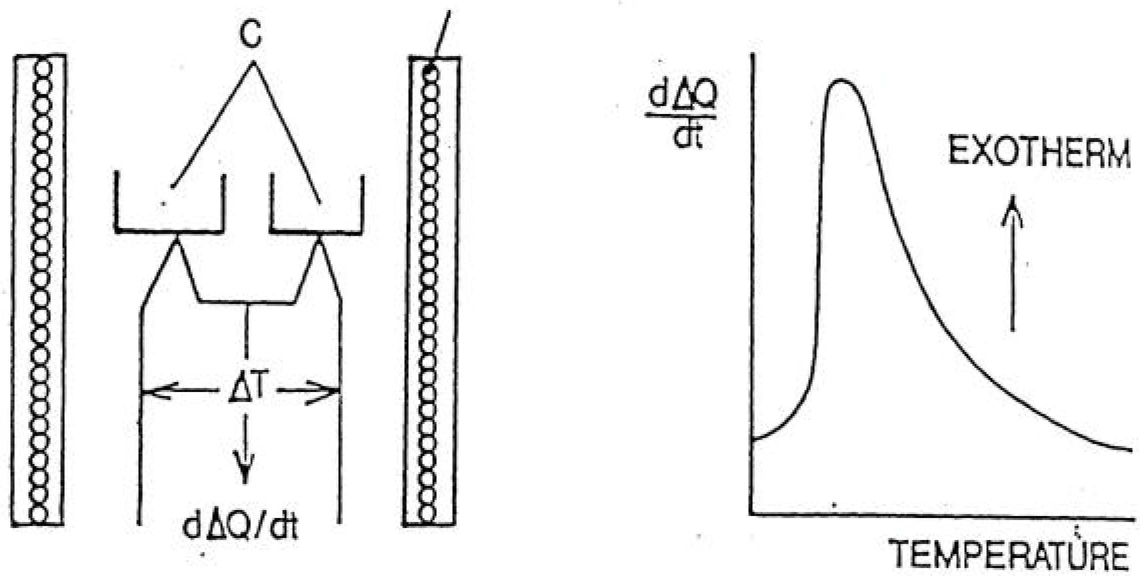
<u>TABLE LEGEND</u>

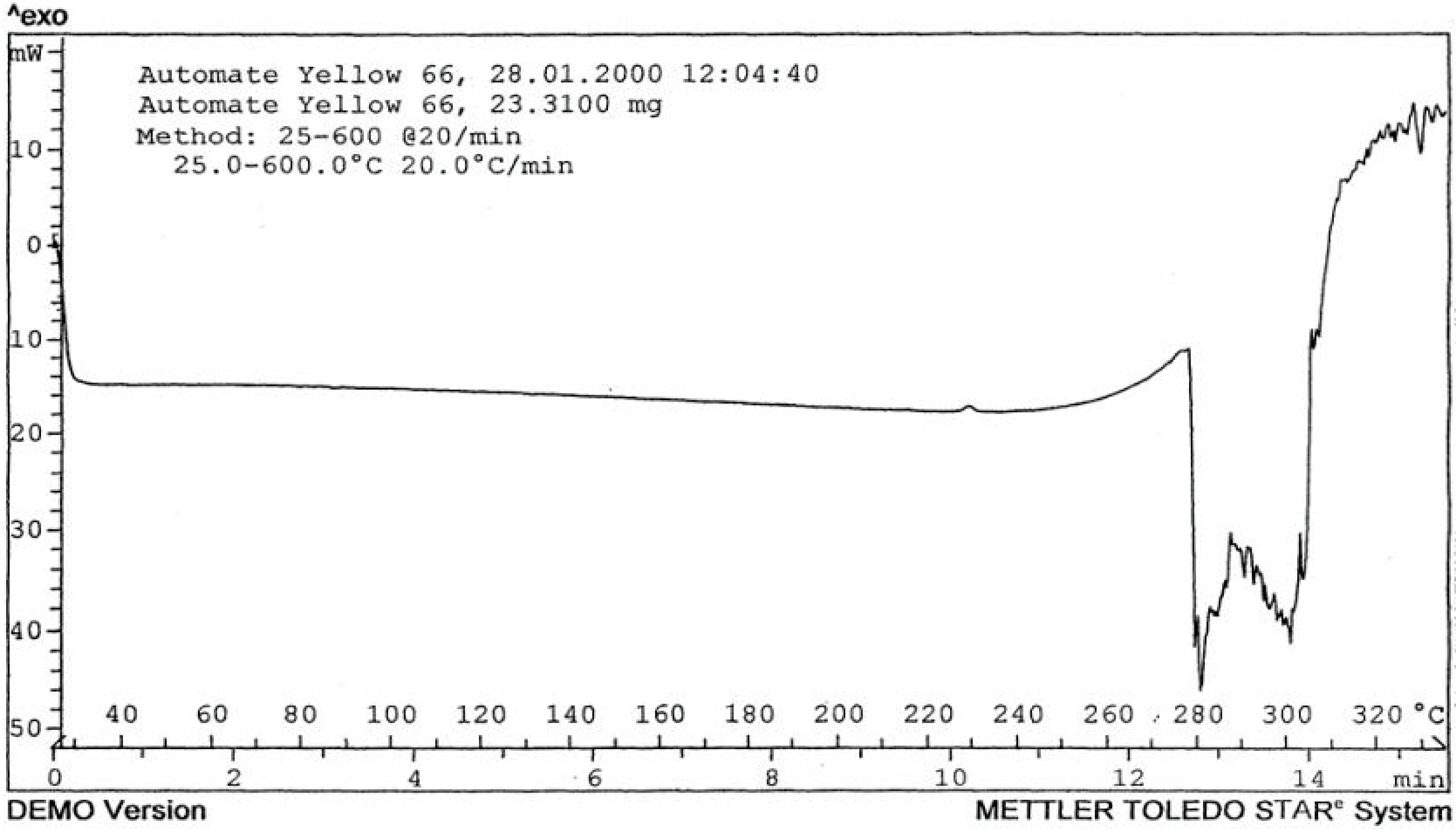
N/A	Not Applicable to current sample, no endotherm observed
T_{START}	Temperature at which the test was started
T_{END}	Final Temperature at test end
T_0	Onset Temperature of corresponding exotherm or endotherm

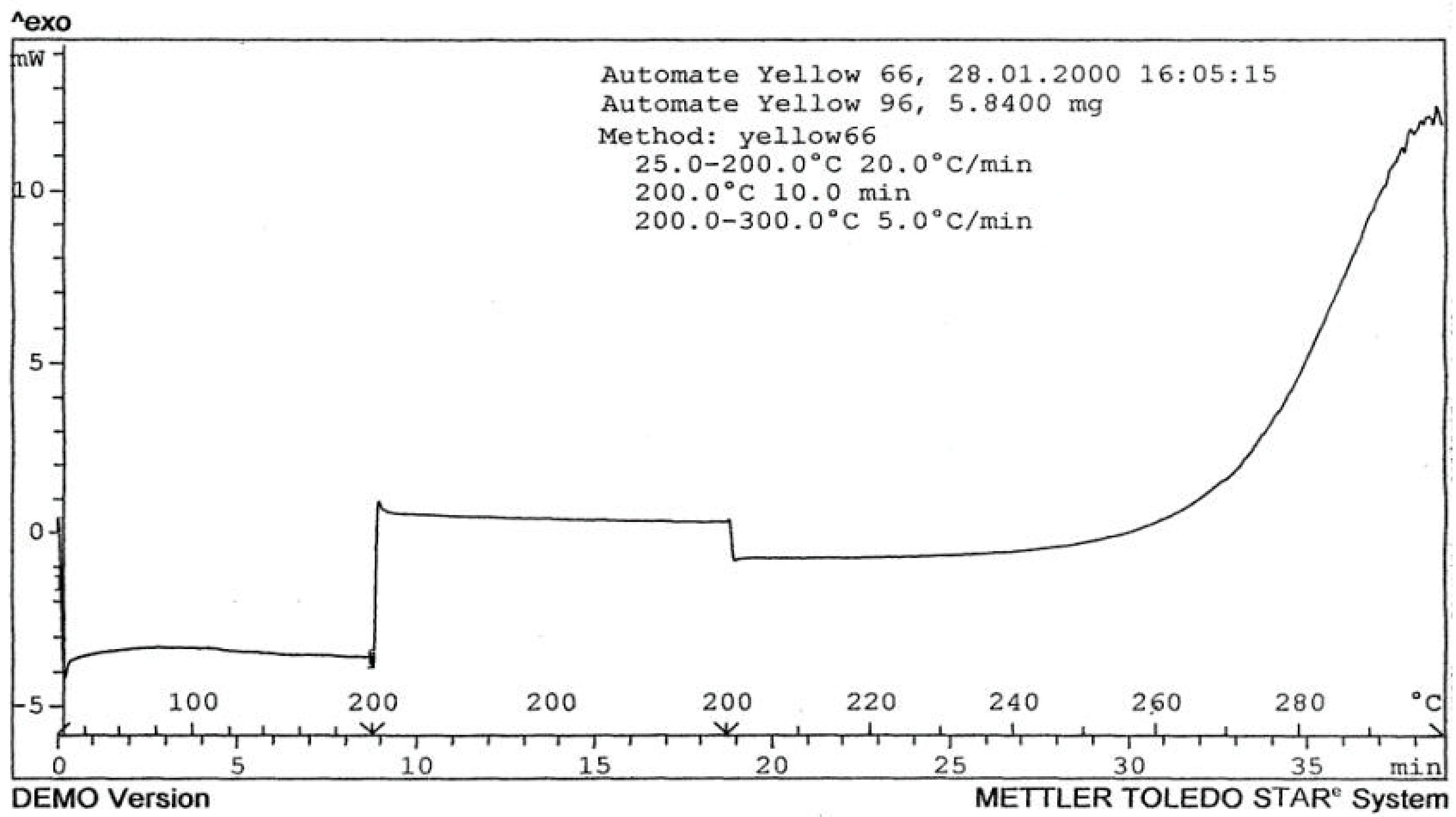
5. DISCUSSION

Following NFPA 704 (Identification of the Fire Hazards of Materials) Automate Yellow 96 is given a reactivity rating of 1 because it produces an exotherm on the DSC having an onset temperature between 150° C and 300° C.

Figure 1: DSC Apparatus and Experimental Trace




^exo
mW
Automate Yellow 66, 28.01.2000 12:04:40
Automate Yellow 66, 23.3100 mg
Method: 25-600 @20/min
25.0-600.0°C 20.0°C/min
10
0
10
20
30
40
50
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 °C
0 2 4 6 8 10 12 14 min
DEMO Version
METTLER TOLEDO STARe System


^exo
mW
10
5
0
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Automate Yellow 66, 28.01.2000 16:05:15
Automate Yellow 96, 5.8400 mg
Method: yellow66
25.0-200.0°C 20.0°C/min
200.0°C 10.0 min
200.0-300.0°C 5.0°C/min
100
200
200
200
220
240
260
280
°C
0
5
10
15
20
25
30
35
min
DEMO Version
METTLER TOLEDO STAR^e System